ASD-TDR-7-911 (V)

IMPROVED PRODUCTION OF POWDER METALLURGY ITEMS

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Basic Industry Branch
Manufacturing Technology Laboratory
Aeronautical Systems Division
Air Force Systems Command
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Wright-Patterson Air Force Base, Ohio

ASD Project Nr 7-911

The synthesizing results on TZM alloy are summarized, and it is shown that it is difficult to obtain the precise carbon content. The atomizing technique has been modified so that superalloy powders of low gas content can now be made on a commercial scale. The results of extrusion experiments in which T-sections are made from PH 15-7Mo alloy, TZM alloy, and a sintered powder billet of Udimet 700 alloy are described and discussed.

(Prepared under Contract AF 33(657)-9140 by IIT Research Institute, Chicago, Illinois, K. Farrell, S A Spachner, and N. M. Parikh).

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FOREWORD

This Interim Technical Documentary Progress Report covers the work performed under Contract AF 33(657)-9140 from 1 July 1963 to 30 September 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with the IIT Research Institute, Chicago, Illinois, was initiated under Manufacturing Methods Project 7-911, "Improved Production of Powder Metallurgy Items." It is being accomplished under the technical direction of Mr. G. W. Trickett of the Basic Industry Branch, ASRCTB, Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Dr. N. M. Parikh of the Institute's Metals and Ceramics Research is the engineer in charge. Others who cooperated in the research and in the preparation of the report were Dr. K. Farrell, Associate Metallurgist; and Dr. S. A. Spachner, Senior Metallurgist. This report has been given the internal designation IITRI-B247-15.

The primary objective of the Air Force Manufacturing Methods Program is to develop on a timely basis manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. The program encompasses the following technical areas:

> Rolled Sheet, Forgings, Extrusions, Castings, Fiber and Powder Metallurgy; Component Fabrication, Joining, Forming, Materials Removal;Fuel, Lubricants, Ceramics, Graphites, Nonmetallic Structural Materials; Solid State Devices, Passive Devices, Thermionic Devices.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

ABSTRACT - SUMMARY
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IMPROVED PRODUCTION OF POWDER METALLURGY ITEMS

I. INTRODUCTION

This is the fifth quarterly report on the subject program, and covers the work done in the period July 1 to September 30, 1963. The fourth quarterly report discussed the results of extrudability tests on wrought or cast superalloy material, and also included an assessment of some pilot-plant superalloy powders. It was concluded that some of the powders would be acceptable for the purpose of the program if their gas contents were found to be low. The present report chronicles the subsequent developments on the superalloy powders, and describes the results of some extrusion trials on sintered superalloy powder billets.

II. RESULTS AND DISCUSSION

A. Synthesized TZM Alloys

One facet of the production of sintered TZM alloy powder preforms, for subsequent forging and extrusion purposes, is the development of TZM powders from a base molybdenum powder. The technique adopted is to add the necessary titanium and zirconium alloying additions to the molybdenum in the form of hydrides. After a ball-milling treatment to insure a good, random distribution of the hydrides in the molybdenum, the mixture is heated in argon at 600° C to reduce the hydrides. Fine graphite powder is then added, and the mixture again ball-milled, then compacted and sintered at 2000° C for 2 hours in vacuo. Chemical analyses showed that this technique gave the required titanium and zirconium contents (0.5wt% and 0.1 wt%, respectively), but the carbon content was consistently below the specification of 0.02 wt%. Also, oxygen contents were high. It was thought that carbon was being lost as an oxide during sintering. Accordingly, the graphite additions were stepped up to compensate for the carbon losses. The carbon contents of a number of sintered TZM compacts with different initial graphite additions are given in Table I.

TABLE I

CARBON CONTENTS OF SINTERED TZM ALLOY POWDER COMPACTS AS A FUNCTION OF INITIAL GRAPHITE ADDITION

Initial Graphite Addition, wt%	Final Carbon Content, wt%
0. 1	0. 0061
0. 3	0. 0057
0. 5	0. 0064
0. 6	0. 07
0. 7	0. 06
0. 8	0. 12
1. 0	0. 51
0. 52	0. 09
0. 52	0. 06
0. 55	0. 11
0. 55	0. 14
0. 58	0. 05
0. 58	0. 13
0. 40	0. 01
0. 50	0. 01

The earliest results are placed in the top section of the table and would seem to indicate that the required graphite addition for a residual carbon content of 0.02% should lie between 0.5 and 0.6%. However, the carbon analyses on graphite mixes within this range were all higher than 0.02%. Therefore, graphite additions of 0.4% and 0.5% were tried. The resulting carbon contents were below the specifications. Difficulty here may be encountered in assessing the reliability of the carbon analyses, but other weightier factors must be considered. It is clear that if carbon is lost as an oxide during sintering, then the residual carbon content will be a function of the initial oxygen content of the powder compact. But this oxygen content might vary depending on the molybdenum particle size, shape, and distribution, on the degree of ball-milling given to the powder, and perhaps even on the humidity at the time of preparation. The ball-milling procedure is controlled, of course; but since -325 mesh molybdenum powder is used, there is no control over particle size and distribution. Consequently, the new surface area made available during ball-milling will vary considerably, as will the resultant gas adsorption. Many other variables may interfere, which, even if they could be controlled on a laboratory scale, would present enormous difficulties when the synthesizing technique was upscaled to pilot plant or bulk production level.

It may be advisable, therefore, with this particular alloy, made in this manner, to produce a bulk batch of the powder and to accept whatever carbon content is obtained, every effort being made, of course, to obtain 0.02%. Should the alloy fall short of or exceed the carbon specification it should still be forged and extruded, and its mechanical properties should be assessed in terms of the analyzed carbon content. Should these properties prove satisfactory, further effort could then be devoted towards the improvement of chemical control during the synthesizing process.

B. Atomized Powders

Gas analyses are now available on the Hoeganaes pilot plant superalloy powder compacts made from argon-melted stock. Previous gas analyses had indicated that these powders might be low in oxygen but, as was pointed out in the fourth quarterly report (ASD-TDR-7-911 (IV)), there were marked discrepancies between the analyses from two different laboratories, and caution was urged in judging the results until a third laboratory had been consulted. In Table II, the results on sintered compacts of -80 mesh powder from this third laboratory (C) are compared with those from the more reliable of the two former laboratories (B).

Although there are differences in the oxygen results from the two sources, it is obvious that the oxygen contents exceed the arbitrary maximum limit of 0.1 wt%, and do not differ greatly from those of the sintered powder compacts prepared from prealloyed melt stock (Table III). To further highlight the difficulties encountered in obtaining agreement between laboratories on chemical analyses, it can be seen that the nitrogen contents listed by Laboratory C are, in two cases, about nine times greater than the corresponding figures from Laboratory B.

The above powders were thought to be unsuitable because of their excessive gas contents. Therefore, the atomizing technique was modified, at some expense, to produce powders of lower gas content. A pilot-scale melt of Udimet 700 (melt No. AH 133) was atomized under the new conditions. Gas analyses of -80 mesh powder by Laboratory B gave 0.047 wt% oxygen and 0.0013 wt% nitrogen. These figures are very good indeed, and are well within the acceptance limits of gas content.

This latest powder is somewhat coarser than previous powders and has a higher apparent density and better flow rate, but it has one slight drawback. The individual particles tend towards a more rounded shape than previous powders, and hence have poorer "keying" properties and much inferior green strengths. However, experiment has indicated that this shape factor can be overcome by changing one of the variables in the atomizing process. This change should result in more irregular shaped powders without increase in gas content. Since we know from our earlier work that atomization does not alter the over-all chemical composition of the superalloys, we feel we are now in a position to make bulk quantities of high-quality superalloy powder for the forging and extrusion preforms.

TABLE II

GAS CONTENTS OF FULLY SINTERED, PILOT PLANT ATOMIZED POWDER COMPACTS PREPARED FROM ARGON-MELTED STOCK

		Laboratory B		Laboratory C	
Alloy	Melt. No.	Oxygen, wt%	Nitrogen, wt%	Oxygen, wt%	Nitrogen, wt%
Inco 713C	H346	0. 160	0. 0058	0. 134	0. 044
Udimet 700	H348	0. 133	0. 0048	0. 152	0. 041
PH 15-7Mo	H344	0. 250	0, 0012	0. 182	0. 001

TABLE III

GAS CONTENTS OF FULLY SINTERED, PILOT PLANT ATOMIZED POWDER COMPACTS PREPARED FROM PREALLOYED MELT STOCK

		Laboratory B	
Alloy	Melt No.	Oxygen, wt%	Nitrogen, wt%
Inco 713C	H345	0. 136	0. 0010
Udimet 700	H347	0. 125	0. 0025
PH 15-7Mo	H342	0. 210	0. 0015
PH 15-7Mo	H343	0. 180	0. 0060

C. Hot-Working Tests on Superalloy Compacts

Some further hot-working tests have been made on 1 in. high x 1 in. diameter sintered superalloy powder compacts. These compacts were made from the Hoeganaes argon-melted stock powders which we now know to have high oxygen contents. The compacts were hot-rolled at about 1200°F (650°C) but, being small, they cooled very rapidly. The PH 15-7Mo compact was rolled without difficulty to 1/8 in. plate in six passes. This might indicate that larger PH 15-7Mo powder billets should not prove difficult to extrude. Our experience with the extrusion of large wrought billets of PH 15-7Mo lends confirmation to this view. The Inco 713C compact collapsed after several rolling passes (and much plastic deformation), while the Udimet 700 compact broke into several pieces on the first rolling pass. The two latter alloys may be extremely sensitive to deformation temperature, or they may have cracked because of their oxide contents. Microexamination of the three compacts after the rolling treatment showed oxide stringers in all cases, and recrystallization in PH 15-7Mo and Inco 713C alloys. The microstructures were much the same as those shown for the press-forged compacts in ASD-TDR-7-911 (IV).

D. Extrusion Activity

1. General

Although rod extrusion experiments during the previous reporting period produced a relatively small amount of sound rod, some definite conclusions could be reached by comparison of extrusion data obtained and from the appearance of the extruded product. Ram force vs. stroke and ram velocity vs. stroke curves, billet temperature, and extrusion condition indicated that:

(a) Billet temperature monitoring equipment would occasionally read 200°-300°F low

(b) PH 15-7Mo stainless steel and TZM alloy are not strain-rate sensitive, and can be extruded at ram speeds as high as 600 in/min.

(c) Successful extrusion of U-700 (Udimet 700) and Inco 713C alloys requires relatively low ram speeds, and close control of billet temperature

This extrusion information appeared sufficient to establish guidelines for production of shaped extrusions, assuming temperature monitoring problems could be solved. Consequently, extrusion activity during this reporting period was concerned with a determination of optimum extrusion parameters for the production of T-sections of 1/4 in. web, circumscribed by a 2 in. diameter circle. Wrought PH 15-7Mo stainless steel, wrought TZM alloy, and sintered powder U-700 alloy extrusion billets, available during this quarter, were used for this study.

2. Accurate Temperature Monitoring

During the early part of 1962, Ray-O-Tube measurement of extrusion billet temperature was substituted for thermocouple temperature measurement technique. Reasons for this were as follows:

(a) It was determined that a thermocouple in contact with the side of the extrusion billet, under light pressure, would read 250°-300°F low at a temperature of 2250°F. A thermocouple bead peened into a small hole in the billet would read 100°-150°F low at 2250°F. A spot-welded thermocouple bead would give an accurate reading.

(b) Since welding of a thermocouple to the billet was obviously impractical during an extrusion run, the only other possibility of thermocouple measurement lay in measurement of the base temperature of the billet by insertion of the thermocouple in the refractory pedestal of the induction furnace. This procedure was not employed, because it did not appear likely that the base of the extrusion billet, in contact with the furnace refractory, would be the same temperature as the rest of the billet. Consequently, a decision was made to monitor temperature by a non-contact device.

(c) A Ray-O-Tube (Leeds and Northrup Co. total radiation gage) operated in conjunction with an AZAR recorder proved to be accurate in this application. Sighting into a closed furnace refractory, "black body" conditions could be obtained regardless of emissivity coefficient of billet material or its glass coating. Maintenance of an argon flow between Ray-O-Tube sight port and the billet surface prevented temperature measurement errors due to billet or glass coating fumes. A fixed billet-to-gage distance assured reproducibility of temperature readings.

Since this device had operated reliably for many months, the recent demonstrated inaccuracy of temperature measurement was believed to be due to an equipment breakdown, rather than improper measurement technique. Investigation proved this to be the case. Furnace refractory cracking and crumbling, coupled with a softening of the silica connecting tube between the Ray-O-Tube housing and the refractory, had caused a partial obstruction of radiation striking the Ray-O-Tube radiation reflecting mirror, with a resultant loss of temperature-monitoring accuracy.

The difficulty was remedied by replacement of the furnace refractory in the 80 kw induction coil, and installation and alignment of a new quartz connecting tube. There have been no further instances of billet overheating since this repair was effected.

3. T-Section Extrusion of PH 15-7Mo Stainless Steel

First extrusion trials produced T-sections with less than 10° twist per linear foot, but did cause breakage of the one-piece zirconia-coated dies used. Breakage always occurred at the end of one or more of the webs, permitting metal to extrude into the crack. This, in turn, would develop a raised line on the end of the web. Examination of the broken dies showed that die fracture was due to inadequate support of the die body. Since the "T" dies are placed inside the liner in the present tooling setup, shrink rings could not be placed around the die.

Use of a three-piece die assembly appeared to be a likely solution to the problem. Such a die would not break under high extrusion stress, and could be zirconia-coated and ground at a considerably lower cost than a single-piece die. Accordingly, the one-piece dies on hand were modified to a three-piece configuration. Modification consisted of:

(a) Placement of a shim between each die segment. Shim thickness was equal to that of the metal removed, and was ground flush with the die coating.

(b) A milled groove on the die OD to permit wiring of the die together for handling ease.

(c) Three pins fitted into the die base to prevent segment sliding during handling.

The three-piece dies performed well. Metal die bodies were undamaged by extrusion, and could be reused. Die coatings were generally good for only one trial, however, in contrast to rod die coatings, which had a life of 2 to 6 extrusions.

Some difficulty was encountered in maintaining proper placement of the shims. Shims tended to move and/or wear slightly during extrusion, causing a light scoring of the ends of the T-webs. Use of molybdenum in place of steel shim stock did not help to correct this difficulty. Consequently, shim use is being discontinued. Instead, die segments are being coated on their sides as well as on the face. Side coatings on each segment are then ground to half the present shim thickness. It is believed that this procedure will eliminate extrusion scoring, and increase ease of handling during insertion of die into container.

Five T-sections of this alloy have been produced as required. Sections are reasonably straight, and possess a good surface finish. Optimum extrusion parameters were found to be as follows:

Billet temperature	2000°F
Ram speed	400-500 in/min
Billet lubricant	Corning glass No. 0010
Container lubricant	Fiskelube 604
Peak extrusion pressure	140,000 psi

Graphite pusher blocks were used on all extrusions. Billet-and-block transfer time, in all cases, was less than 10 seconds.

4. T-Section Extrusion of TZM Alloy

The extrusion ratio of the die used for all T-section extrusion efforts is 13.4:1. Extrusion of TZM rod at a 16:1 ratio had shown that:

(a) Minimum extrusion temperature was 3300°F. Extrusion resistance of alloy beging to decrease markedly above 3450°F. At 3600°F, extrusion force is only 65% of that required at 3300°F.

(b) TZM alloy is not strain-rate sensitive, and may be extruded at ram speeds of 600 in/min. Relatively high ram speeds reduce billet-liner-die contact time, and minimize tooling damage.

Extrusion pressure required for a 13.4:1 extrusion ratio of asymmetrical shape could be expected to be at least as great as that required for a 16:1 ratio extrusion possessing uniaxial symmetry. Consequently, an extrusion temperature of 3400° F was selected for the first T-section trial. Unfortunately, accumulator pressure was marginally low at the stroke, with the result that only 162,000 psi was available for breakthrough pressure. (Normally accumulator will develop 180,000 psi on stem.) Although the billet did extrude at 162,000 psi, the ram speed obtained was considerably less than the preset speed. As a result, ram speed averaged only 125 in/min, instead of the desired 600 in/min. Approximately 3 1/2 in. of billet extruded, producing a 40 in. T-section, before the billet chilled to a point where it stalled the ram.

The extrusion produced possessed relatively little twist, and had a smooth surface. However, considerable tool damage was sustained. The lead-in section of the extrusion die was melted, and the upper "T" shear blade badly washed. One section of the T-extrusion jammed so tightly in the shear bolster that machining was required to remove it.

Although this particular extrusion trial was destructive to the tooling, it did establish the maximum time available for TZM extrusion when billet is at 3400° F. In this case, the billet was under extrusion pressure in the container for approximately 7 seconds before the ram stalled. Seven seconds represents the maximum time for extrusion of a 3 1/2 in. diameter billet.

It is believed that reduction of extrusion time from 7 seconds to 1 second, while maintaining all other extrusion parameters at the same values, will solve the problem of TZM T-section extrusion. Extrusion parameters used were as follows:

Billet temperature	3400° F
Ram speed	100-200 in/min
Billet lubricant	Corning glass No. 7740
Container lubricant	Alpha Molykote
Peak extrusion pressure	162,000 psi

A graphite pusher block was used. Billet-and block transfer time was less than 10 seconds.

5. T-Section Extrusion of Sintered Powder U-700 Alloy

Two U-700 alloy sintered powder billets were used for this study. This particular material had an oxygen content of 0.125%, considerably higher than that of material currently being produced. Extrusion of this material was attempted because it was believed that extrusion data could be developed which would be useful in extrusion of the higher quality material, and it was the only powder alloy billet material available at the time.

Prior attempts at extrusion of cast U-700 alloy to rod at a 12:1 extrusion ratio had shown that:

(a) Extrusion temperature range was extremely narrow, probably between 1900°F and 2100°F.

(b) Ram speed would have to be held to less than 90 in/min to prevent gross edge cracking or extrusion breakup.

These two extrusion conditions work against one another. If the billet is not extruded promptly, it will stiffen and stall the ram. If the billet is extruded quickly, the extrusion is not sound.

Since high-speed extrusion was out of the question, some means had to be developed for reducing the billet heat transfer rate. Two procedures were employed. First, a plasma-arc coated alumina liner was used. The alumina coating possessed a considerably lower coefficient of thermal conductivity than that of tool steel. Secondly, the billet was encased in a 1/16 in. thick mild steel can. This can was spun on the billet. Clearance between the billet and can was on the order of a few thousandths of an inch. No top was welded on this can. Its sole purpose was to prevent immediate chilling of the wall of the U-700 alloy billet when the billet was placed in the extrusion container. (Stainless steel was initially considered because of its lower thermal conductivity, but was rejected because of the difficulty in acid-etching stainless steel from U-700 alloy without attacking the U-700.)

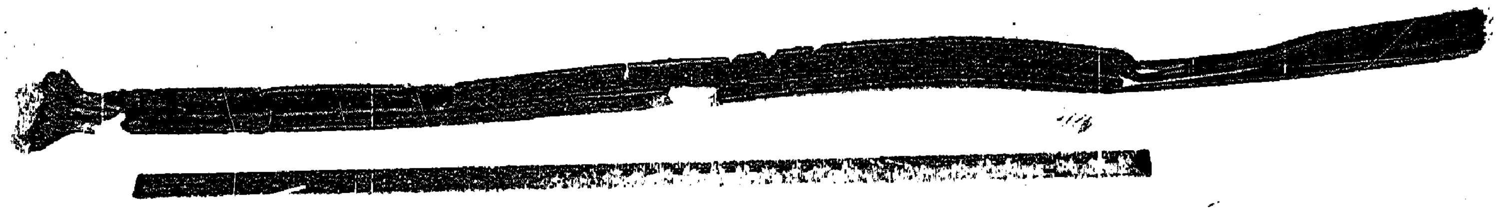
Mild steel leader blocks were used to facilitate entry into the "T" die. Front ends of both steel leader block and billet were nosed at a 45° angle. Rear end of the leader block was bored out at a 135° angle to accommodate the 45° billet nose.

A billet temperature of 2050°F was employed for the first extrusion trial. Approximately 1 inch of billet extruded before the ram stalled at a 152, 000 psi stem pressure. A second trial was made with accumulator pressure increased to develop a peak stem pressure of 180, 000 psi, and a billet temperature of 2100°F. This time, the billet extruded completely, producing a 50 in. long T-section. This T-section was sound for the first 6 in. Progressive side cracking occurred past this point, however, which eventually caused tearing of the extrusion. A photograph of this extrusion is shown in Figure 1. Even though the extrusion has many fractures, sound metal areas may be found along the full length, and in all webs of the T-section. Microstructures of a sound area are shown in Figures 2 (longitudinal section) and 3 (cross-section). The grains are small and equiaxed, but evidence of the extrusion direction is given by the oxide stringers.

Comparison of the extrusion force vs. stroke curve and ram velocity vs. stroke curve with the extrusion produced showed that the ram velocity varied between 60 and 80 in/min over the stroke. Where ram velocity was 60 in/min over the first 1/2 in. of stroke, the extrusion was sound. When ram velocity exceeded this value, the extrusion showed cracking which increased in depth as ram velocity increased. Required stem pressure was surprisingly low, once breakthrough was achieved. Breakthrough pressure was 160, 000 psi, running pressure about 115, 000 psi. Conclusions drawn from this information are as follows:

(a) It appears likely that a reduction of ram speed by 20 in/min will eliminate the edge cracking observed.

(b) The relatively low running pressure indicates a possibility of reducing billet temperature by 25°F, to further reduce any tendency toward hot shortness.



Neg. No. 25552

Fig. 1

Photograph of T-Section extruded from a sintered powder billet of Udimet 700 alloy

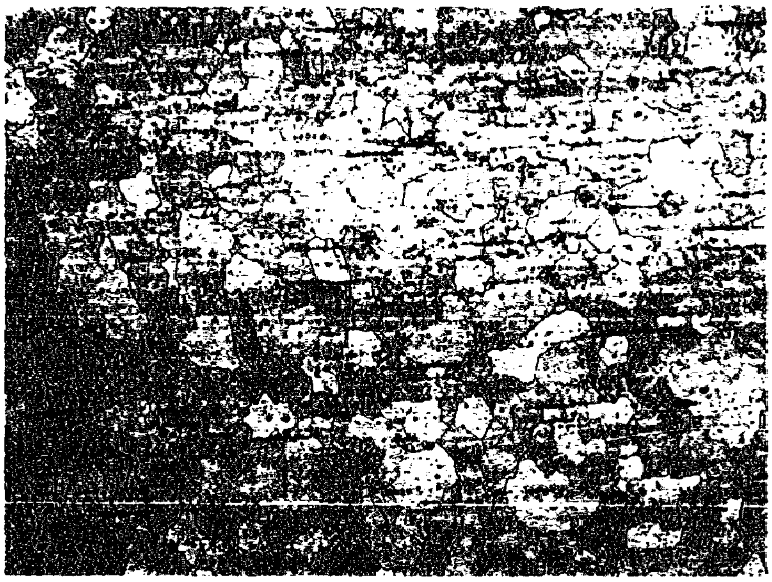


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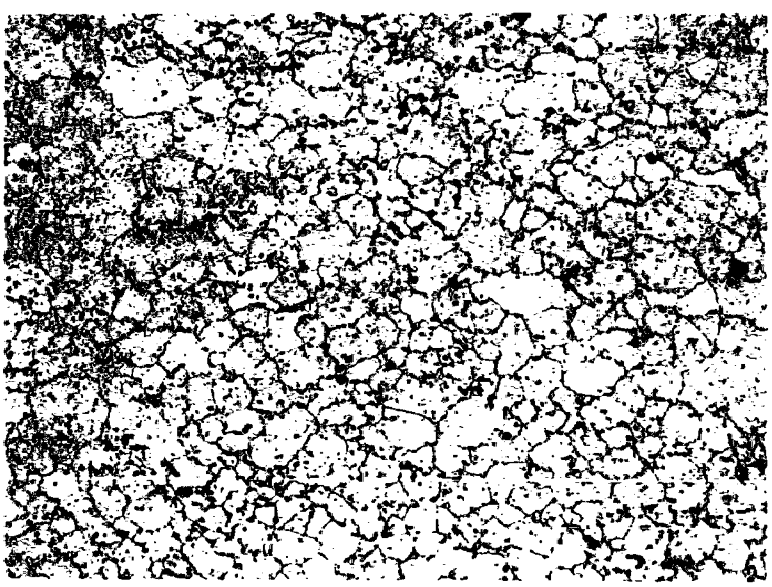
Fig. 2

Microstructure of longitudinal web of T-section extruded from sintered Udimet 700 alloy powder



Neg. No. 25607 X200

Fig. 3

Microstructure of cross-section of T-section extruded from sintered Udimet 700 alloy powder

(c) The rapid drop in pressure after breakthrough is achieved indicates that the configuration of the billet nose is not optimum. This need not be changed immediately, however, since breakthrough is presently being effected with moderate stem pressure.

Successful adjustment of the extrusion parameters in the manner indicated should make shaped extrusion of sintered powder U-700 alloy billet highly probable. The effort will be facilitated by the use of presently available powder containing 60% less oxide than that used for this study.

III. CONCLUSIONS AND FUTURE WORK

It is concluded that:

(a) In synthesized TZM alloy it is proving difficult to obtain carbon contents exactly on the specification of 0. 02%.

(b) Superalloy powders atomized from argon-melted stock have oxygen contents in excess of the acceptance limit of 0. 1%.

(c) Modifications to the atomizing technique now give superalloy powders with oxygen contents of 0. 05%.

(d) Hot rolling tests on superalloy powder compacts of high oxygen content show that the ease of deformability follows the order (1) PH 15-7Mo, (2) Inco 713C, (3) Udimet 700, with PH 15-7Mo being quite ductile and Udimet 700 brittle.

(e) T-sections have been extruded from wrought PH 15-7Mo alloy and from TZM alloy.

(f) A T-section has been extruded from a sintered powder billet of Udimet 700 alloy of high oxygen content.

The results on the superalloy powder of low gas content and on the extrusion work are very encouraging. To our knowledge Udimet 700 alloy has never before been extruded to a shape other than a round. We feel that this success, though not yet complete, proves the soundness of the powder metallurgy approach to making superalloy shapes. We have shown that the process is feasible on the alloy which proves the most resistant to hot working. Future work will be devoted to producing good

T-sections from all the superalloys once permission is obtained to purchase bulk quantities of the low-oxygen powders.

IV. LOGBOOKS AND PERSONNEL

The experimental data on the project are recorded in Logbook No. C-12789. The personnel contributing to the program are C. J. Carter (Assistant Metallurgist), K. Farrell (Associate Metallurgist), S. A. Spachner (Senior Metallurgist), and N. M. Parikh, Scientific Advisor.

Respectfully submitted,

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